

## **NASA Technical Memorandum 100770**

# **Determination of Fiber Volume in Graphite/Epoxy Materials Using Computer Image Analysis**

**Michael J. Viens**

**November 1990**



National Aeronautics and  
Space Administration

Goddard Space Flight Center  
Greenbelt, MD

(NASA-TM-100770) DETERMINATION OF FIBER  
VOLUME IN GRAPHITE/EPOXY MATERIALS USING  
COMPUTER IMAGE ANALYSIS (NASA) 11 p

CSCL 110

N91-12767

Unclas

G3/24 0318165



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**Michael J. Viens**  
*Goddard Space Flight Center*  
*Greenbelt, Maryland*



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# Determination of Fiber Volume in Graphite/Epoxy Materials Using Computer Image Analysis

## ABSTRACT

The fiber volume of graphite/epoxy specimens was determined by analyzing optical images of cross sectioned specimens using image analysis software. Test specimens were mounted and polished using standard metallographic techniques and examined at 1000 times magnification. Fiber volume determined using the optical imaging agreed well with values determined using the standard acid digestion technique. The results were found to agree within 5% over a fiber volume range of 45 to 70%. The error observed is believed to arise from fiber volume variations within the graphite/epoxy panels themselves. The determination of ply orientation using image analysis techniques is also addressed.

## INTRODUCTION

The determination of fiber content in graphite/epoxy materials is required to accurately predict the mechanical and physical properties of composite structural members. The traditional technique used to determine fiber volume is by means of acid digestion. This technique requires that the cured graphite/epoxy material first be dissolved in boiling nitric acid and then washed in acetone. The environmental impact of the acid digestion technique is twofold. The fumes generated during digestion may not be completely captured in the condenser. The acid digestion technique also produces over 200 milliliters of waste chemicals for each gram specimen.

Optical examination is an alternate way to evaluate the fiber volume. Test specimens are cross sectioned and polished, then analyzed using an optical microscope. The limitation of the optical technique has been that the imaging programs were available only on large computers or that the laboratory techniques available were tedious and required extensive operator interaction.

Recent improvements in both personal computers, and imaging software and hardware have made the use of the imaging techniques a viable alternative to acid digestion.

To verify the accuracy of the imaging technique and establish an image evaluation procedure, several specimens of graphite/epoxy material which had previously had the fiber volume determined using the acid digestion technique were evaluated using image analysis. The verification of laminate ply orientation is also investigated. This paper reports the results of the testing performed and recommends techniques to standardize fiber volume analysis.

## EXPERIMENTAL PROCEDURE

### Test Specimens

The test specimens used in this study were acquired from various ongoing, in-house projects. All of the material evaluated was taken from panels manufactured using prepreg material. The prepreg materials used and the associated specimen ID's are listed in Table 1. The fiber (or ply) orientations given are taken from the normal to the cross section surface. These are the orientations intended during the manufacture of the panels and subsequent specimen preparation.

The variety of prepreg specimens acquired enabled the evaluation of fiber volumes

Table 1. Fiber Volume Specimens

| Specimen ID | Prepreg Material    | Fiber Orientation (degrees) |
|-------------|---------------------|-----------------------------|
| 1,2         | Fiberite T300/934   | 0                           |
| 3           | 3M SP500 T40-12k    | 0                           |
| 4           | Hercules AS4/3501-6 | 0                           |
| 5           | Hexcel T300/F-155   | 0                           |
| 6,7,12,13   | Fiberite T50/934    | 0                           |
| 8,9,14,15   | Fiberite T50/934    | 45                          |
| 11,17       | Fiberite T50/934    | 0,30                        |
| 10,16       | Fiberite T50/934    | 0,60                        |

ranging from 45 to 70%. While the acid digestions reported here were performed by various individuals, all of the digestions were performed according to ASTM D3171 (Test Method for Fiber Content of Resin Matrix Composites by Acid Digestion).

The specimens examined optically were taken from areas directly adjacent to those of the acid digestion specimens. The size of the optical specimens was generally on the order of the acid digestion specimens. The area of the cross sections examined varied from 12 to 24 square millimeters. The optical specimens were mounted in room temperature curing epoxy and subsequently polished to reveal the graphite fibers.

### Test Equipment

The image analysis was performed with an imaging program entitled "NIH Image" (version 1.29). This software is public domain shareware which was created at the National Institute of Health. The program was run on an Apple Macintosh IIfx computer. Images were acquired directly from a microscope (Nikon Epiphot) via a video camera and a frame grabber, image acquisition board (Quick Capture, Data Translation, Marlboro MA).

All images acquired in this study were taken at 1000 times magnification. Preliminary studies revealed that the fiber edges could be resolved only at the greatest available magnification. A typical image is shown in Figure 1.

### Image Acquisition

Four basic tasks are performed to determine fiber volume. (1) The subject area is selected and brought into focus. (2) The image is then captured and is corrected for variations in the light intensity using a "Blank Field" image. The blank field correction is also a background correction that removes artifacts on the image created by any dust on the optical lens. (3) The fiber and resin areas are partitioned using a "threshold" operation, which changes the fiber area to black and the resin to white. (4) The number of black and white pixels is then measured. The ratio of

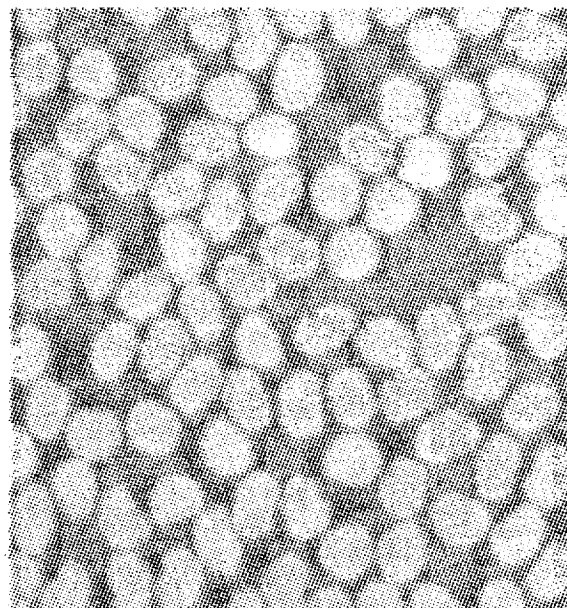


Figure 1. Cross section of specimen 6. The fiber orientation is normal to the surface. Fiber diameters are approximately 5 microns

black pixels to the total number of pixels in the interrogated area is the percentage of fiber area.

The assumption made at this point is that the fiber area percent is equal to the fiber volume percent. The adoption of this assumption is not intended to imply that the fiber volume of the entire panel can be determined by the evaluation of a two dimensional slice through a small portion of the panel. What the assumption does imply is that the fiber area percent is a good approximation of the fiber volume percent in the vicinity of the cross section. The information gained at the cross section must be correlated to some other physical condition of the panel. This other physical condition might be thickness or ultrasonic impedance. It should be noted that the acid digestion technique requires a similar assumption. The acid digestion technique evaluates only a small portion (one gram) of the entire panel (1000 gram).

### Thresholding

Of the four tasks described above to determine the fiber volume the most important is the thresholding operation. The threshold value must be selected in a



consistent manner. The criteria used to select the threshold value must eliminate operator bias and minimize the effect of slight variations in the fiber/resin distribution. It was found in the course of this study that the threshold value could be selected by using the distribution of pixels as a function of gray level. A typical histogram of gray scale versus number of pixels is shown in Figure 2.

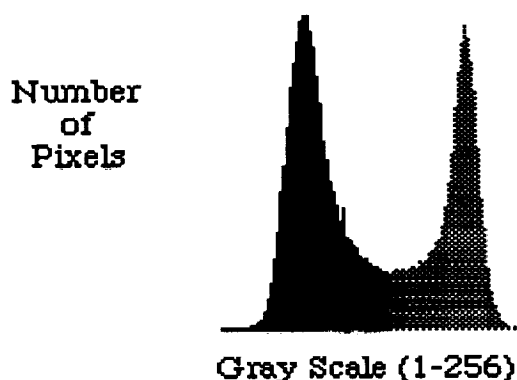


Figure 2. Histogram taken from the Figure 1 image. Peak on the left represents graphite fibers and the peak on the right represents the resin matrix.

The threshold value was selected by locating the minimum "number of pixels" value between the fiber and the resin peaks. The gray level that corresponded to this minimum value was selected for the threshold value. It was found that this value would shift slightly from frame to frame by not more than 2 or 3 gray levels in either direction. The average threshold gray level for five frames was used as the threshold value for the remaining samples. To insure that the threshold value selected was appropriate for each frame, the histogram with threshold level high lighted was quickly examined during the analysis of each frame. It was determined that a 5 gray level difference in threshold value would change the fiber area measured by 1%.

It was found that the intensity of the reflected light would vary with fiber orientation. The fibers cross sectioned at larger angles were more reflective. This variation in light intensity also varies the threshold value for each fiber orientation. The specimens with several ply orientations required separate

thresholds for each fiber orientation. The threshold adjustment is easily accomplished but does add to the analysis time.

Preliminary studies indicated that the analysis of fibers in the 90 degree orientation (fibers oriented parallel to the specimen surface) would yield consistently greater fiber area than the other orientations. Figure 3 is a typical histogram of a 90 degree ply taken from the same panel as specimen 6.

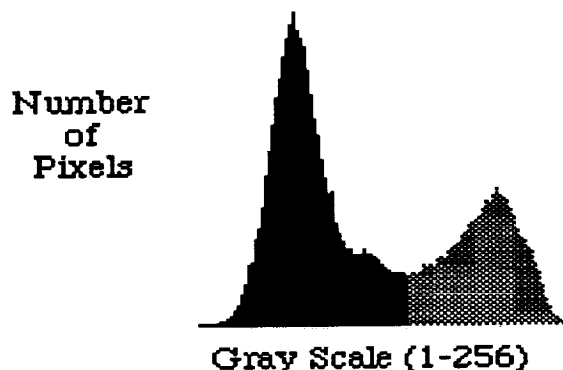


Figure 3. Histogram of 90 degree ply.

The shape of the 90 degree fiber peak tends to be slightly distorted. Note the slight shoulder on the fiber peak. The resin peak also tends to be lower and wider. The higher resin contents determined by the 90 degree fiber orientation are probably due to the portion of the fibers just below the resin surface contributing to the fiber peak. The subsurface fibers raise the intensity of the reflected light causing broadening of both the fiber and resin distribution. The subsurface portion of the 0, 30, 45 and 60 degree fibers was not optically visible.

## RESULTS AND DISCUSSION

### Fiber Volume Analysis

To analyze an entire 24 mm<sup>2</sup> area at 1000 times magnification would require a total of 1500 frames to be acquired. If the system were totally automated this might be reasonable but because the present system requires manual stage movement and

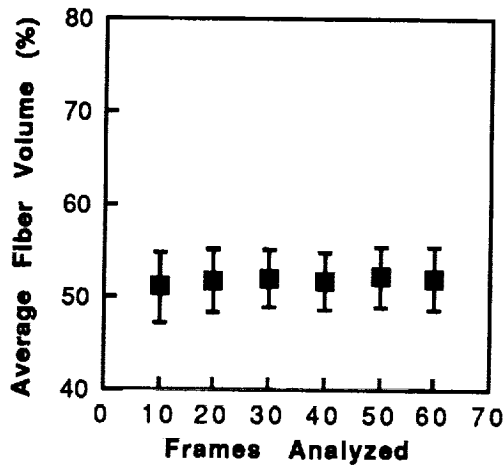


Figure 4. Fiber volume of specimen 17 as a function of samples taken.

focusing, the number of frames acquired and analyzed must be somewhat less.

Figures 4 and 5 show the mean and standard deviation of specimens as a function of the number of video frames analyzed. Specimen 17 (Figure 4) had an even distribution of fibers and very little change in mean or standard deviation values were noted with increased sampling.

Specimen 5 (Figure 5) had large variations in fiber density from frame to frame. The fiber areas ranged from 35 to 70% of the total area. This specimen also had significant a number of voids. It was decided that 50 frames were adequate to produce a good estimate of fiber area.

Figure 6 shows the average fractional fiber area determined from the optical image analysis plotted versus fiber volumes determined using the acid digestion technique. The values are also listed in Table 2 along with the standard deviation of fiber areas from the optical technique. The optical results were generally within one standard deviation of the digestion results. Specimens 10 and 16 were not included in the regression. It was noted that the digested portion of specimen 10 had a large

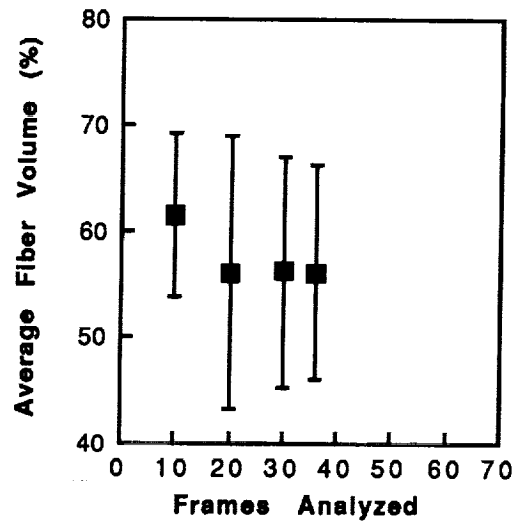


Figure 5. Fiber volume of specimen 5 as a function of samples taken.

Table 2. Fiber Volume Results

| ID | Fiber Vol Digestion | Fiber Vol Imaging | Std Dev Imaging | Frames Analyzed |
|----|---------------------|-------------------|-----------------|-----------------|
| 1  | 64.00               | 64.34             | 2.99            | 30              |
| 2  | 64.50               | 62.75             | 2.39            | 20              |
| 3  | 70.55               | 73.47             | 1.82            | 30              |
| 4  | 71.70               | 71.13             | 5.04            | 20              |
| 5  | 55.10               | 56.10             | 10.10           | 36              |
| 6  | 53.80               | 53.77             | 3.23            | 50              |
| 7  | 55.90               | 59.17             | 2.04            | 50              |
| 8  | 53.00               | 52.22             | 2.98            | 50              |
| 9  | 52.40               | 51.95             | 3.20            | 50              |
| 10 | 63.00               | 50.95             | 3.36            | 50              |
| 11 | 45.82               | 47.61             | 3.15            | 50              |
| 12 | 52.13               | 52.85             | 2.19            | 50              |
| 13 | 49.91               | 48.81             | 2.24            | 50              |
| 14 | 46.17               | 48.85             | 3.96            | 50              |
| 15 | 59.90               | 57.85             | 1.94            | 50              |
| 16 | 47.99               | 55.15             | 3.52            | 58              |
| 17 | 49.60               | 51.90             | 3.41            | 60              |

depression. This depression could have been created during the manufacture of the panel by a foreign object which subsequently became detached. The result would be an area with a concentration of fibers.

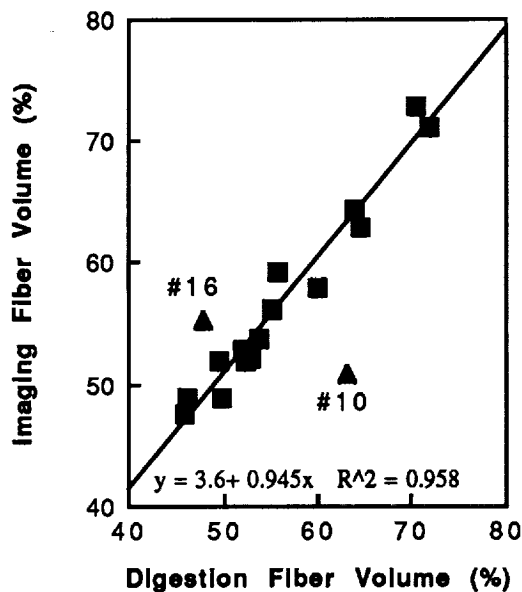


Figure 6 Fiber volume determined using the imaging technique versus the digestion technique. Triangles indicate specimens which were not used in the regression (10 and 16).

It was noted during acid digestion of the Fiberite T50/934 panels that the fiber volume varied by more than 10%. It is felt that the portion of specimen 16 evaluated optically had a greater concentration of fibers than the adjacent portion which was digested.

A further confirmation of this is shown in Figures 7 and 8. The optical specimen thickness correlates well with the fiber content (Figure 7). However, the optical specimen thicknesses of specimens 10 and 16 do not correlate as well to the acid digestion fiber volumes (Figure 8).

#### Fiber Orientation Analysis

Assuming that a graphite fiber is an ideal, round cylinder, a cross section normal to the fiber axis is a circle. A slice at any angle, other than normal or parallel to the fiber axis, is an ellipse. The ratio of the minor and major axes is the cosine of the angle at which the slice is taken.

The imaging program used in this study has the capability to count particles and measure

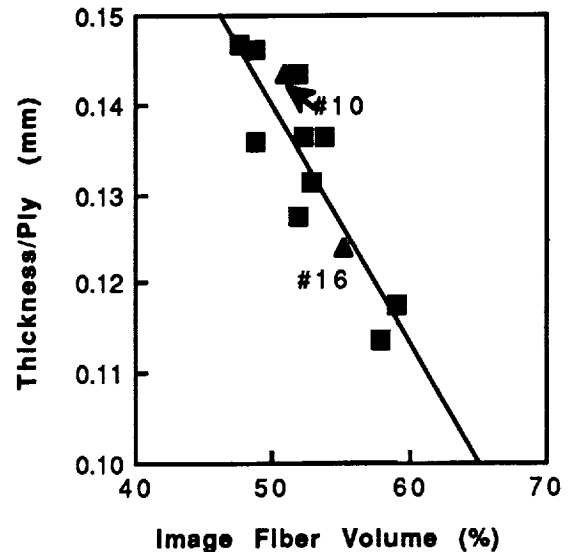


Figure 7. Thickness per ply of the Fiberite T50/934 optical specimens versus the fiber area. Note the agreement of specimens 10 and 16.

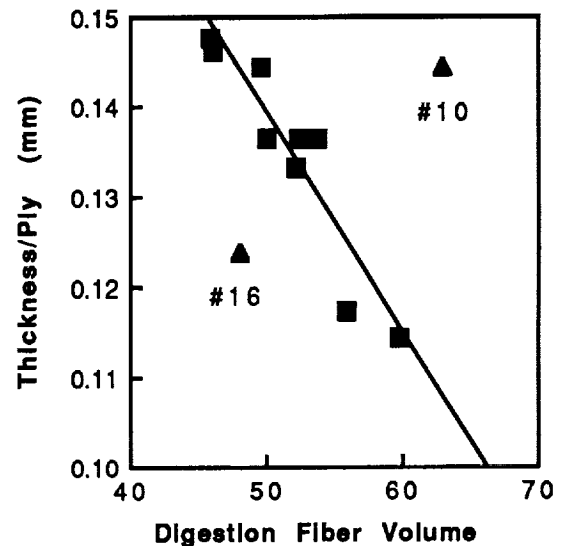


Figure 8. Thickness per ply of the Fiberite T50/934 optical specimens versus the fiber volume of the adjacent acid digestion specimen.

their minor and major axes and particle area. The fibers were treated as particles and the particle size analyzed was restricted such that only individual fibers were detected (i.e. fibers which do not touch any other fibers).

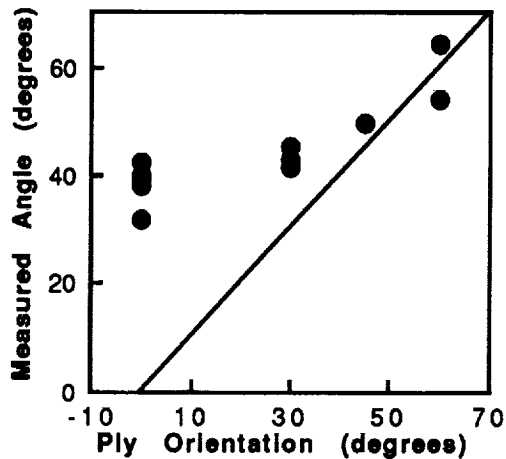


Figure 9. Angle of fibers measured optically using ratio of ellipse axes plotted versus the angle of the ply intended during manufacture. Curve is theoretical one to one correlation between the two techniques.

Table 3. Optical Evaluation of Ply Orientation

| ID | Lay-up Angle (degrees) | Measured Angle (degrees) | Average Area (pixels) |
|----|------------------------|--------------------------|-----------------------|
| 6  | 0                      | 42.4                     | 853                   |
| 11 | 0                      | 39.1                     | 791                   |
|    | 30                     | 45.2                     | 928                   |
| 13 | 30                     | 42.8                     | 875                   |
|    | 0                      | 38.0                     | 858                   |
| 14 | 45                     | 49.8                     | 1242                  |
| 16 | 0                      | 31.7                     | 813                   |
|    | 60                     | 53.8                     | 1244                  |
|    | 60                     | 64.1                     | 1764                  |
| 17 | 0                      | 39.9                     | 829                   |
|    | 30                     | 41.5                     | 983                   |

The fibers touching the edge of the frame are also ignored.

The calculation of fiber angle based on only the ratio of major to minor axes assumes that the fibers are regular cylinders. This is not often the case with small graphite fibers. The fibers in Figure 1 are oriented normal to the surface. The calculated angle for these fibers should be zero but the image analysis of the major and minor axes indicated that the fibers are oriented at a 42 degree angle to the surface.

The fiber cross section angle for several fiber orientations was analyzed and is presented in

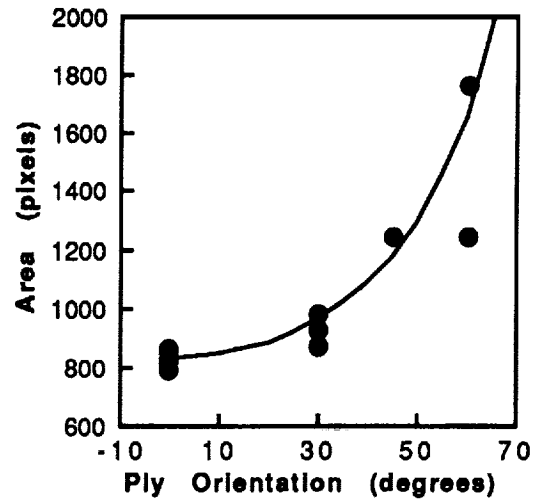


Figure 10. The average area of a single fiber is plotted versus the intended angle of the ply. The curve through the data is the average area of the zero degree fibers divided by the angle cosine.

Table 3. Specimens 11, 16 and 17 had several fiber orientations. As shown in Table 3 and Figure 9 the error decreased as the angle increased.

An alternate method to determine fiber angle is to evaluate the area of the fibers. The ratio of the circle and the ellipse areas also equals the cosine of the slice angle. The measured areas are listed in Table 3 and plotted versus ply lay-up orientation in Figure 10. The line through the data is the average area of the zero degree fibers divided by the angle cosine.

The results of specimen 16 indicate that at least one of the 60 degree plies is off the lay-up angle. The low zero degree area indicates that the cross section was taken approximately normal to that ply. The large difference in both area and measured angle for the 60 degree plies indicates that at least one of these plies was not oriented correctly during the manufacture of the panel.

It is not clear that one of these methods is superior to the other in determining fiber orientation. The area technique requires an area value for the zero degree fibers as a benchmark. It is possible that a laminate lay-

up is such that a zero degree cross section of the fibers is not easily obtained. The axis ratio technique appears to be accurate for large angles (>45 degrees) but for small angles large errors are observed.

It is important to note the limitations in the determination of the fiber orientation. The fiber orientation at the specimen cross section surface is dependent on the mounting of the specimen. It may be more appropriate to evaluate the relative fiber orientation than an absolute orientation.

It should also be noted that the evaluation of ply angle can not determine the sense of a ply. A ply oriented at a plus 60 degrees will appear to have the same orientation as a ply oriented at minus 60 degrees.

### SUMMARY

This test program has shown that the fiber volume percent of graphite/epoxy laminates can be estimated using imaging techniques and the results agree with those obtained by acid digestion tests. The image analysis technique also provides an indication of the fiber distribution on a microscopic scale.

The local fiber volume can be correlated to an easily measured physical attribute such as thickness. This correlation allows the determination of fiber volume percent over the entire panel or component.

### ACKNOWLEDGEMENTS

The author would like to thank Timothy Van Sant for his extensive support with the imaging software and Bradford Parker for his help in acquiring the test specimens. Metallographic specimen preparation was performed by Patricia Friedberg.

# Report Documentation Page

|   |  |  |  |   |  |
|---|--|--|--|---|--|
| 1. Report No.<br>NASA TM-100770   |  | 2. Government Accession No.                              |  | 3. Recipient's Catalog No.  |  |
| 4. Title and Subtitle<br>Determination of Fiber Volume in Graphite/Epoxy<br>Materials Using Computer Image Analysis   |  |  |  | 5. Report Date<br>November 1990   |  |
|   |  |  |  | 6. Performing Organization Code<br>313  |  |
| 7. Author(s)<br>Michael J. Viens  |  |  |  | 8. Performing Organization Report No.<br>91B00019                                     |  |
|   |  |  |  | 10. Work Unit No.   |  |
| 9. Performing Organization Name and Address<br><br>Goddard Space Flight Center<br>Greenbelt, MD 20771   |  |  |  | 11. Contract or Grant No.   |  |
|   |  |  |  | 13. Type of Report and Period Covered<br>Technical Memorandum                         |  |
| 12. Sponsoring Agency Name and Address<br><br>National Aeronautics and Space Administration<br>Washington, DC 20546-0001  |  |  |  | 14. Sponsoring Agency Code  |  |
|   |  |  |  |   |  |
| 15. Supplementary Notes<br><br>M. Viens is affiliated with the Goddard Space Flight Center, Greenbelt, Maryland.  |  |  |  |   |  |
| 16. Abstract<br><br>The fiber volume of graphite/epoxy specimens was determined by analyzing optical images of cross-sectioned specimens using image analysis software. Test specimens were mounted and polished using standard metallographic techniques and examined at 1,000 times magnification. Fiber volume determined using the optical imaging agreed well with values determined using the standard acid digestion technique. The results were found to agree within 5% over a fiber volume range of 45% to 70%. The error observed is believed to arise from fiber volume variations within the graphite/epoxy panels themselves. The determination of ply orientation using image analysis techniques is also addressed. |  |  |  |   |  |
| 17. Key Words (Suggested by Author(s))<br><br>Graphite/Epoxy<br>Composite Fiber Volume<br>Computer Imaging  |  |  |  | 18. Distribution Statement<br><br>Unclassified - Unlimited<br><br>Subject Category 24 |  |
| 19. Security Classif. (of this report)<br><br>Unclassified  |  | 20. Security Classif. (of this page)<br><br>Unclassified |  | 21. No. of pages<br><br>10  |  |
|   |  |  |  | 22. Price   |  |